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Application for Patent

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A METHOD FOR PARTICLE SIZE AND CONCENTRATION MEASUREMENT

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A METHOD FOR PARTICLE SIZE AND CONCENTRATION MEASUREMENT

A METHOD FOR PARTICLE SIZE AND CONCENTRATION MEASUREMENT

Field of the Invention

The present invention is related to the field of measuring particle size and concentration. More specifically it relates to the use of optical methods for measuring particle size and concentration. The particles could be liquid borne, airborne or on a surface.

Background of the Invention

Publications and other reference materials referred to herein are incorporated herein by reference in their entirety and are numerically referenced in the following text and respectively grouped in the appended Bibliography, which immediately precedes the claims.

Many techniques exist for particle size and concentration analysis (PSA), they can be reviewed for reference in the book by Terry Alan (1) "Introduction to Particle Size Analysis". The most commonly used techniques are optical, based on the interaction of the measured particles with laser radiation. Especially when approaching the particle size range

around 1 micron and below, most of these techniques suffer from inaccuracies due to Mie diffraction and the effect of the real and imaginary part of the particle's refractive index. It is known, for example, that in some techniques, such as techniques based on Fraunhoffer diffraction analysis, light absorbing particles would be over sized due to energy loss resulting from the absorption, while in high concentration, particles would be under sized due to secondary scattering etc.

An optical technique that is less sensitive to this problem is known as Time of Transition or TOT. In this technique the interaction of a scanning, focused laser beam and the particles is analyzed in the time domain rather than in the intensity domain, resulting in lower sensitivity to variation in the refractive index. A detailed description of the technique appears in the paper (2) by Bruce Weiner, Walter Tscharnuter, and Nir Karasikov. To a great extent, in this technique, a de-convolution algorithm, of the known laser beam profile, from the interaction signal, derives the size. The concentration is derived from the number of interactions per unit time within the known volume of the focused laser beam.

The interaction of the particles in the TOT technique is with a focused scanning laser beam. In order to measure smaller particles, a smaller focused spot should be used. However according to diffraction laws for a

Gaussian laser beam, if the beam's waist is D, the divergence of the beam is proportional to λ/D where λ is the laser's wavelength. The trade-off between the ability to resolve small particles, to the focus volume and the accuracy in measuring concentration is obvious. Thus if the TOT technique is targeted to resolve and measure particles in the micron range it would be limited in its ability to measure low concentrations as the instantaneous focus volume is small and the interaction rate of particles is low. On the other hand, taking a larger spot will improve the concentration measurement but will degrade the quality and resolution of the size analysis.

An improvement could be achieved by using a shorter wavelength. This could have a limited effect of, as high as, a factor of 2 only, since going to too short a wavelength will result in absorption of the laser light by the optics and, in the case of particles in liquid, also absorption by the liquid.

It is therefore the purpose of the present invention to introduce a new technique and means to decouple between the two contradicting requirements: the ability to resolve small particles and the ability to measure low concentration.

Further purposes and advantages of this invention will appear as the description proceeds.

<u>Summary of the Invention</u>

The present invention is a novel method for particle size and concentration analysis. The technique is applicable for liquid borne particles; airborne particles and particles on a surface. The particles size range that can be measured with the method of the invention is from sub-micron to thousands of microns. The ability to decouple focus dimensions from the depth of focus is used for PSA, offering a high resolution and better concentration sensitivity at low concentrations. A special embodiment of the invention addresses the implementation for high concentration.

Provisions for an adaptive range, for triggering "legal" interactions and for the detection of smaller particles in dark field TOT mode are also part of the present invention.

The method of the invention is based on synthetic beam generation, which can give a factor 15 of improvement over the diffraction limit case in the level of concentration that could be measured for a known size range. By implementing this method, fine particles can be analyzed for size and their concentration can be measured even at very low levels.

The limitation as described hereinabove in the background results from the inherent Gaussian beam profile of the laser beam. Other energy distributions could be synthetically generated. One specific reference, which describes the technique, is reference (3). This publication deals with the generation of three-dimensional light structures used in the invention. It describes the philosophy, the techniques used, and also provides some examples. In particular, the dark beam described is of primary interest for the present invention. Other relevant references are (4)-(9). The dark beam is a laser beam that has a dark spot at the center of a beam with an otherwise Gaussian envelop. The main advantage of this beam for the purpose of the present invention originates form the fact that the dark central spot is narrower than a classical Gaussian spot leading to the possibility of higher sensitivity to the position and structure of an obstructing object. Dark beams can be generated by converting a conventional laser beam with the help of an optical element (usually a diffractive element) or by a special design of the laser resonator in such a way that it emits a dark beam. These laser modes are usually members of a set called Gauss-Laguere modes.

Dark beams can be generated in such a way that they maintain a sharply defined energy distribution over a wider depth of field, thus offering a better trade-off between size and concentration when implemented in the

TOT technique. A few ways to realize these forms could be considered and are covered in the references listed in the bibliography.

The use of such beams for PSA is part of the present invention where:

- The beam is adapted in focal spot size and depth of focus to the size and concentration range.
- The detection of the scattered light is based on the energy profile extracting the size and concentration information in the most efficient way.
- Provisions for overcoming ambiguous measurements in high concentration are an inherent part of the invention.

Another aspect of the invention is the use of the TOT or the enhanced TOT, described in the present invention, to measure particles in very high concentrations.

The present invention provides a method of particle size and concentration measurement comprising the following steps:

- providing a focused, synthesized, non-Gaussian laser beam:
- causing the beam to interact with the particles;
- measuring the interaction signal and number of interactions per unit time of the beam with the particles; and

- using algorithms to map the interaction signals to the particle size and the number of interactions per unit time to the concentration.

The particles can be suspended in a fluid, airborne, or on a surface and their size can range from submicron to thousands of microns. The focal properties of the laser beam are changed depending on the size and concentration range of the particles.

The focused, synthesized, non-Gaussian laser beam can be a dark beam and the measurements are made in the intensity domain or by using the time of transition (TOT) technique.

According to the method of the invention, the non-Gaussian beam can be generated by employing a mask over a Gaussian laser beam. The Gaussian beam is spatially modulated by use of, for example, a spatial-filter, a set of spatial filters, an electronic spatial light modulator, or a liquid crystal device. The spatial modulation can comprise intensity modulation, alternating intensity modulation, polarization modulation, and combinations of these.

In preferred embodiments of the invention, the non-Gaussian beam is generated by directly modifying the laser cavity or combining the beams from several lasers.

The interaction of the focused beam with the particles is accomplished either by causing the particles to flow relative to a stationary beam or by providing a scanning mechanism that provides a linear or a rotary scanning path for the focused beam.

In a preferred embodiment of the invention, a detector is used to measure radiation scattered at 90 degrees to the beam direction to verify single particle interaction in the focal area. This detector can be, for example, a CCD camera.

In another embodiment of the invention, a detector is used to measure radiation scattered at 90 degrees to the beam direction in order to detect smaller particles using dark field TOT measurement.

In another embodiment of the invention, high concentrations of particles are measured by using a reflection mode, collecting the back-scattered interaction energy from the particle. These measurements can be carried out using TOT.

According to the method of the invention, the algorithms used to map the interaction signals to the particle size and the number of interactions per unit time to the concentration can be either explicitly based on the interaction signals or based on an advanced artificial intelligence machine, such as a Neural Network or support vector machine (SVM).

The present invention further provides a system for particle size and concentration measurement comprising:

- one or more lasers to provide a Gaussian laser beam;
- a scanning mechanism;
- means for converting the Gaussian laser beam into a structured (non- Gaussian) laser beam; and
- detection means.

In a preferred embodiment of the invention, the means for converting the Gaussian laser beam into a structured (non- Gaussian) laser beam consist of a combination of a spatial filter and a lens.

In other preferred embodiments, the system of the invention additionally comprises a second detector to measure the radiation scattered at 90 degrees to the beam direction. The system can comprise a beam splitter to divert back-scattered interaction energy from the particle to the detector.

All the above and other characteristics and advantages of the invention will be further understood through the following illustrative and non-limitative description of preferred embodiments thereof, with reference to the appended drawings.

Brief Description of the Drawings

Fig. 1 schematically shows the beam profile and its shape after interaction with particles;

Fig. 2 schematically shows the layout of the invention; and

Fig. 3 schematically shows layout of the embodiment of the invention used at high concentration.

Detailed Description of Preferred Embodiments

The invention deals with a novel way for particle size and concentration measurement using a laser beam whose energy profile is optimized for the particle's size and concentration range.

A preferred embodiment of the invention involves the use of a dark beam, i.e. a beam with a dark spot in its center. This beam profile in combination with the enhanced depth of focus, which is another feature of the invention, allows operation in low concentrations. Additional information, which exists as both broadening of the main beam and change of the central dark spots, yields information on the particle size. The dynamic

range of measurement is thus extended where smaller particles interact with the dark spot whereas larger particles interact with the main beam.

Fig. 1 illustrates the mechanism. Viewing the light intensity scattered by a very small particle crossing a dark beam with the spatial profile (1), the detector will register a similar pattern in the time domain. However, the interaction signal obtained for a large particle (2) is characterized by the elimination of the dark center and by broadening of the signal. The interaction with an intermediate sized particle (3) maintains the original signal width but decreases the depth of the dark spot. There are thus two major parameters of the signals for measurement – the signal width and the depth of the central dip. The two signal parameters are sensitive in complementary size ranges and hence assist in broadening the measurement range. The beam design of the present invention applies to both beam width and the shape of the dark spot, as well as to the depth of field. There are thus advantages in size dynamic range and in concentration range.

It is seen that when interacting with a particle larger than the beam, the major effect is beam broadening and the disappearance of the central dip; when interacting with a particle smaller than the beam, the main effect is the decrease of the depth of the signal dip. Thus the single beam provides two signal parameters for better coverage of the size range. As the

synthesized beam is not Gaussian, the algorithm for de-convolving the spot is not straightforward and, among other approaches, one based on artificial intelligence is considered - training the system with several mono-dispersed samples.

Two basic approaches are employed in the invention to generate the non Gaussian beams:

1. A hybrid technique employing a mask over a laser Gaussian beam as schematically depicted in Fig. 2. There are several procedures available to construct optical systems, which convert a Gaussian laser beam (4) into a structured beam (7). Fig. 2 depicts one possible embodiment of the invention, where the spatial-filter (6) - lens (5) configuration is a schematic representation of many possible implementations of the light-structuring configuration. Another embodiment of the invention employs a liquid crystal device to accomplish the spatial modulation. The interaction of the laser beam with the particles is accomplished either by causing the particles to flow relative to a fixed beam or by scanning the beam over the particles. The scanning mechanism (11) facilitates either a rotary scanning path (12) or a linear path. It can be realized by many solutions including a wedge prism, an acousto-optic deflector, etc. Examples of light structures that can be used are Bessel beams and singular beams of various orders, either in one dimension or two dimensions. In Fig. 2 the structured beam (7) interacts with the

particles in the focal zone of the beam. The interaction signal is detected by the detector (8). The signals detected at the detector (8) are as described previously in reference to Fig. 1. In other embodiments of the invention, the modulation of the Gaussian beam is, in addition to intensity modulation that is constant in time, alternating modulation, polarization, modulation, or combinations of these.

The Fourier transform representing the energy distribution in the focus could thus be designed for optimal distribution and depth of focus. Configuration with and without the dark spot, described in Fig. 1, could be realized.

2. A fully synthetic beam profile is an alternative to the technique described above in 1. Specific beam profiles are generated by directly modifying the laser cavity or combining the beams from several lasers. One can use scalar beam structuring as well as vector (polarization) assisted structuring.

Other embodiments of the invention are concerned with beam optimization where, for different size ranges, a different spatial filter (6) is used to generate a different beam profile. This offers the optimal choice of size resolution and concentration accuracy. The variable spatial filter (6) could be in the form of a set of filters that are mechanically mounted in

front of the focusing lens. Another possible embodiment is fully electronic, with a spatial light modulator (SLM) that can be used as an electronically controlled spatial filter.

The detection of "legal particles", intercepting the beam in its focal region, becomes more challenging with the enhanced beam profile of the present invention. Obscuration by multiple particles along the extended focus could erroneously be interpreted as a single particle. The invention optionally addresses this by using an additional detector, which is the triggering detector. Referring again to Fig. 2, the detector assembly (8a) is used for the validation of the particles in the focal zone. This detector detects the scattered radiation at 90 degrees to the beam direction and functions as verification to a single particle interaction in the focal area. Only when the detector (8a) verifies a single particle in the measuring zone, is the signal at detector (8) analyzed for size. The field of view of this side detector can be changed so that it matches the focal region. An array detector such as a CCD camera can be used as well.

Whereas the TOT detection is typically performed in bright field, forward detection, by detector (8), in some cases of smaller particles, the side detector (8a) could be used for the sizing. This is a dark field TOT measurement. The advantage is in an enhanced signal to noise ratio and as such, better resolution of smaller particles.

There are other possible embodiments using the dark field detection, which are also part of the present invention. Blocking the zero order forward scattered energy before the detector (8) is an example for such a possible embodiment.

In some cases the stable concentration to be measured is very high, causing enhanced light scattering and multiple scattering. Typical examples are Liposomes with concentration of 10exp13 1/cc; emulsions with concentrations of 10exp9 particles/cc, etc. In these cases the light beam is diffused after just a short path in the sample. The invention addresses this, in the manner schematically shown in Fig. 3, by minimization of the optical path using a thin cell and, more importantly, by using a reflection mode. In this mode the focusing lens (5) is used also to collect the back-scattered interaction energy from the particle (10). The back-scattered energy is diverted via a beam splitter (9) to the detector (8b). The optical path is minimized by designing the focal zone of interaction to be very close to the focusing lens (5). This minimizes the optical path in the scattering media and therefore the multiple scattering, thus enabling operation in high concentration.

The following example is provided merely to illustrate the invention and is not intended to limit the scope of the invention in any manner.

- A laser source is selected to have the required power and wavelength. For very small particles, i.e. < 0.5 micron, shorter wavelength would be selected, typically 488nm. For larger particles a HeNe laser at 632.8 nm or a semiconductor laser in the near IR could be used.
- A beam profile is generated with the required energy distribution and depth of field in accordance to the size and concentration ranges.
- A scanning mechanism for the beam is introduced. The scanning could be acousto-optic deflection; a rotating wedge prism; a rotating, inclined, optical flat or other means of scanning.
- The scanning velocity should be > 10 times the particle velocity.
- An optical set-up, as shown in Fig. 2, with a detector for the bright field and a detector for the dark field is constructed.
- The detectors bandwidth should be >2Vspot/Rmin, where Vspot is the scanning laser velocity in the interaction area and Rmin is the smallest particle to be detected. The detector's sensitivity should comply with the expected signals

in forward and side scattering. Scattering calculations can be found in the book written by Durst, reference (10).

- The bright field detector should have a dynamic range wide enough to withstand the "no particle" bright field signal.
- The detected signals are fed via a digitizing card to a computer for analysis. Digitization rate should be typically 2π times the frequency.
- The signal analysis is conducted according to the method described with reference to Fig. 1. A mapping of interaction signal to size is thus realized.
- The number of interactions per unit time is calibrated either by building a look-up table interpolating between known concentrations measured or explicitly by calculating the instantaneous volume and the total volume covered in a unit time in order to determine concentration.
- Algorithms for mapping signal to size and interaction rate to concentration can be explicitly based on interaction signals as in Fig 1, or based on an advanced artificial intelligence machine such as Neural Network or SVM (support vector machine).

The method of the present invention is applicable to measuring, for example:

- airborne powders dust/pollution;
- particles suspended in liquid, such as polymer beads; and
- high concentration emulsions, such as mayonnaise.

Although embodiments of the invention have been described by way of illustration, it will be understood that the invention may be carried out with many variations, modifications, and adaptations, without departing from its spirit or exceeding the scope of the claims.

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Claims

1. A method of particle size and concentration measurement comprising the following steps:
   - providing a focused, synthesized, non-Gaussian laser beam:
   - causing said beam to interact with said particles;
   - measuring the interaction signal and number of interactions per unit time of said beam with said particles; and
   - using algorithms to map said interaction signals to said particle size and said number of interactions per unit time to said concentration.

2. A method according to claim 1, wherein the particles are fluid borne, airborne, or on a surface.

3. A method according to claim 1, wherein the size of the particles ranges from submicron to thousands of microns.

4. A method according to claim 1, wherein the focused, synthesized, non-Gaussian laser beam is a dark beam.

5. A method according to claim 1, wherein the measurements are made in the intensity domain.

6. A method according to claim 1, wherein the measurements are made using the time of transition (TOT) technique.

7. A method according to claim 1, wherein the focal properties of the laser beam are changed depending on the size and concentration range of the particles.

8. A method according to claim 1, wherein the non-Gaussian beam is generated by employing a mask over a Gaussian laser beam.

9. A method according to claim 8, wherein the Gaussian beam is spatially modulated.

10. A method according to claim 8, wherein the Gaussian beam is spatially modulated by use of spatial-filter, a set of spatial filters, an electronic spatial light modulator, or a liquid crystal device.

11. A method according to claim 8, wherein the spatial modulation of the Gaussian beam is chosen from the group comprising:

- intensity modulation;
- alternating intensity modulation;
- polarization, modulation; and
- combinations of these.

12. A method according to claim 1, wherein the non-Gaussian beam is generated by directly modifying the laser cavity or combining the beams from several lasers.

13. A method according to claim 1, wherein the interaction of the focused beam with the particles is accomplished by causing said particles to flow relative to a stationary beam.

14. A method according to claim 1, wherein the interaction of the focused beam with the particles is accomplished by providing a scanning mechanism that provides a linear scanning path for said focused beam.

15. A method according to claim 1, wherein the interaction of the focused beam with the particles is accomplished by providing a scanning mechanism that provides a rotary scanning path for said focus beam.

16. A method according to claim 1, further comprising the use of a detector to measure radiation scattered at 90 degrees to the beam direction to verify single particle interaction in the focal area.

17. A method according to claim 15, wherein the detector used to measure radiation scattered at 90 degrees to the beam direction is a CCD camera.

18. A method according to claim 1, further comprising the use of a detector to measure radiation scattered at 90 degrees to the beam direction to detect smaller particles using dark field TOT measurement.

19. A method according to claim 1, wherein high concentrations of particles are measured by using a reflection mode, collecting the back-scattered interaction energy from the particle.

20. A method according to claim 19, wherein TOT is used to measure high concentrations of particles.

21. A method according to claim 1, wherein the algorithms to map the interaction signals to the particle size and the number of interactions per unit time to the concentration are explicitly based on said interaction signals.

22. A method according to claim 1, wherein the algorithms to map the interaction signals to the particle size and the number of interactions per unit time to the concentration are based on an advanced artificial intelligence machine.

23. A method according to claim 1, wherein the advanced artificial intelligence machine is a Neural Network or support vector machine (SVM).

24. A system for particle size and concentration measurement comprising:

- one or more lasers to provide a Gaussian laser beam;
- a scanning mechanism;
- means for converting said Gaussian laser beam into a structured (non- Gaussian) laser beam; and
- detection means.

25. A system according to claim 24, wherein the means for converting the Gaussian laser beam into a structured (non-Gaussian) laser beam consist of a combination of a spatial filter and a lens.

26. A system according to claim 24 additionally comprising a second detector to measure the radiation scattered at 90 degrees to the beam direction.

27. A system according to claim 24, additionally comprising a beam splitter to divert back-scattered interaction energy from the particle to the detector.

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LUZZATTO & LUZZATTO
By ע"י

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One Sheet of Drawings
Sheet No. 1

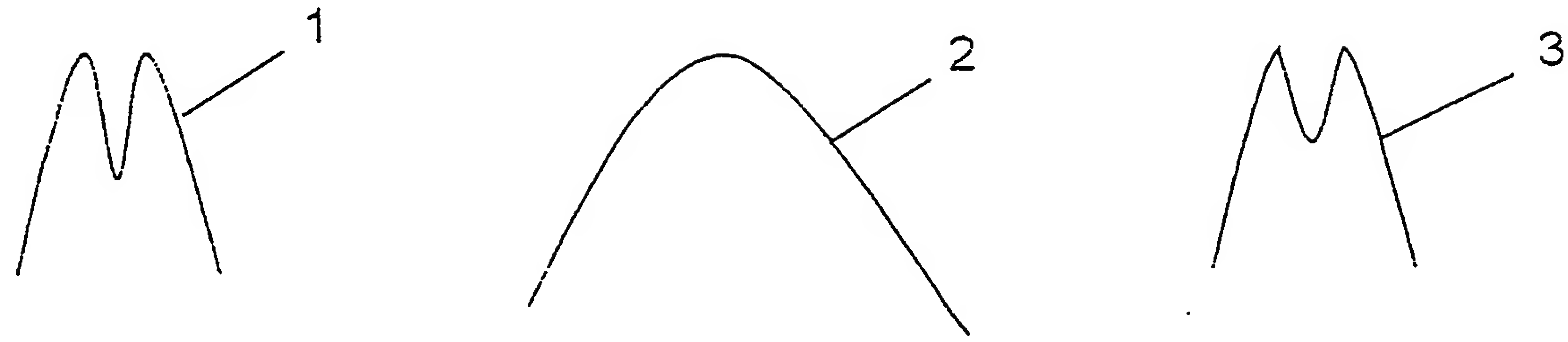

Fig. 1

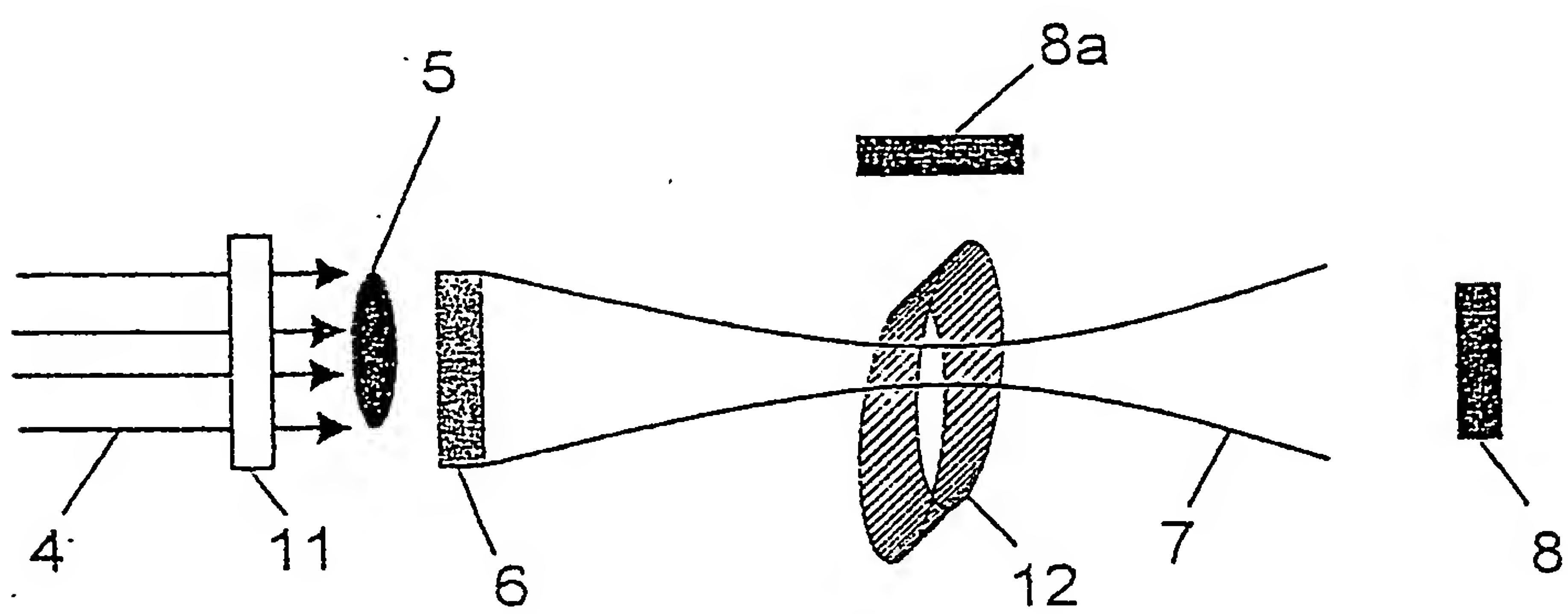

Fig. 2

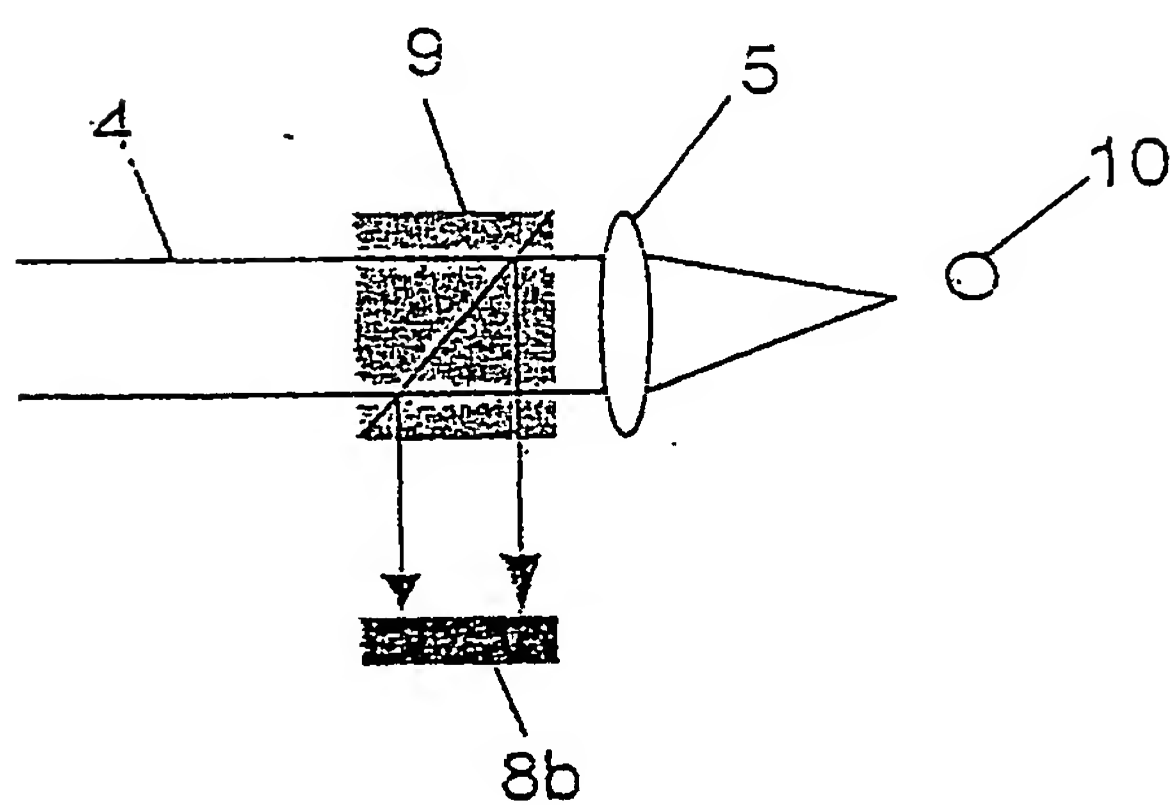

Fig. 3